

ON OPTICAL PROPERTIES AND LUMINESCENCE OF SOME LARGE BANDGAP III-V COMPOUNDS

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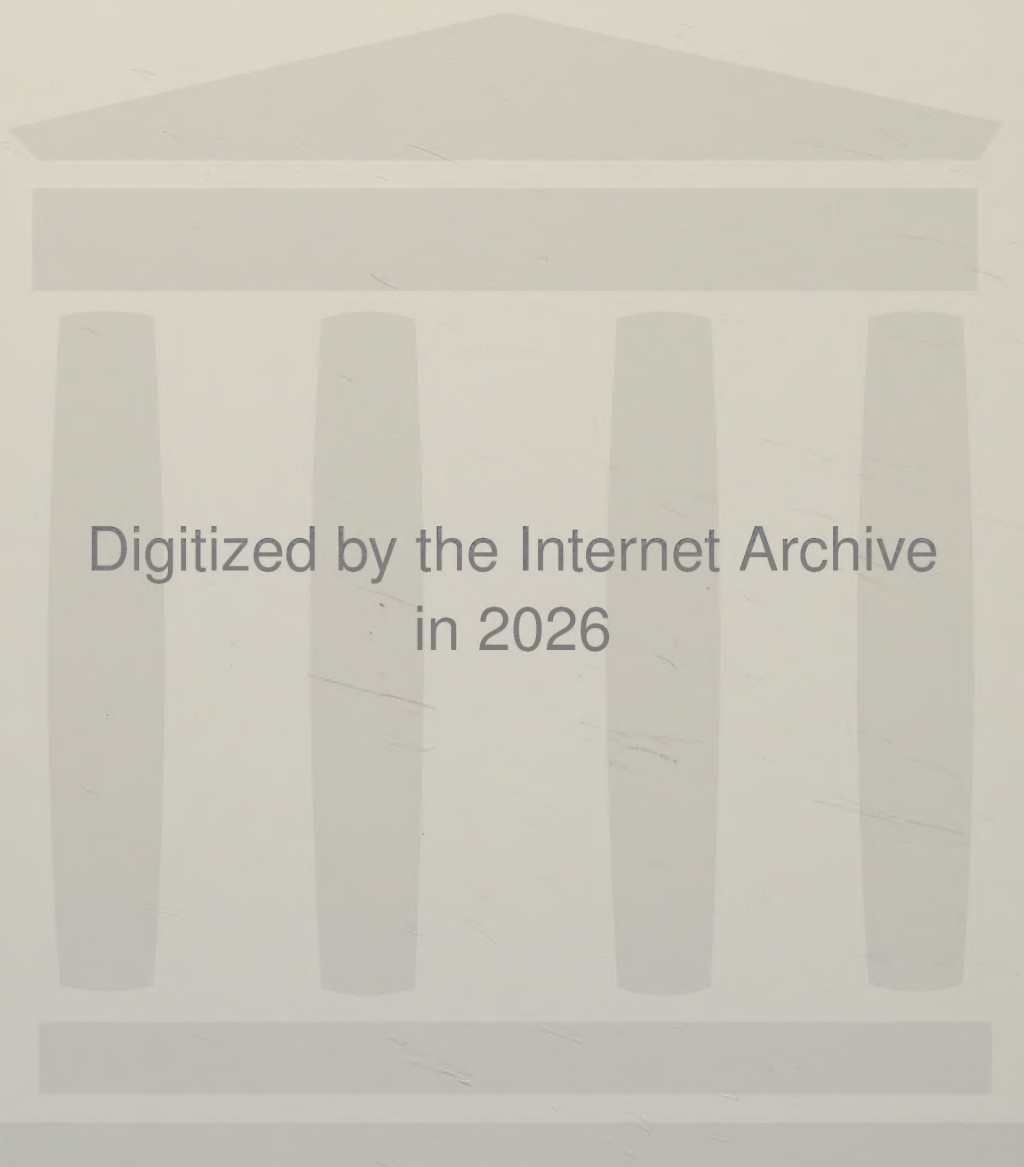


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I INTRODUCTION

Optical phenomena in semiconductors have been a subject for extensive research within the field of solid state physics during the last two decades. Experimental studies of fundamental absorption and reflexion as well as luminescence are the most valuable tools used to obtain reliable information about fundamental optical properties of these materials. However, up to now, the main interest has been focused on the semiconducting elements Si and Ge, some III-V-compounds as GaAs, InSb, GaP, InP, GaSb, AlSb and InAs, and II-VI-compounds as CdS, ZnS, CdSe, CdTe, ZnTe, ZnSe, HgTe etc. The general rule has been, that materials, which are relatively easy to prepare for important technical applications, have collected the main interest for physical investigations, while other materials, which have some unattractive physical or chemical properties, or which could not be prepared by conventional methods, were set aside for the future.

Thus there are still a few simple inorganic semiconductors, the physical properties of which are rather poorly explored. Among the III-V-compounds, GaN, AlP and AlAs belong to this group. The reasons why GaN has been very little investigated might be, that the preparation of crystals of convenient size by conventional methods was unsuccessful, and thermal dissociation was found to occur already at rather low temperatures¹⁻³⁾. New techniques for crystal growth developed for other III-V-compounds in the last years, however, have now been found to be applicable on GaN^{4,5)}, and thus the interest in this compound seems to have mounted during the last year. Further GaN is regarded as a very promising material for many applications, e g electroluminescent devices.

A similar situation is present for AlP and AlAs. The main reason

for the low interest in these compounds was their instability in moist air^{6,7)}, which made them unattractive for applications. These large bandgap semiconductors, however, are also of a great interest for potential optical devices in the visible spectral region, and therefore it might be possible to overcome the difficulties in preparation and handling for these compounds.

II THE AUTHOR'S CONTRIBUTION

The work done by the author with respect to physical properties of GaN, AlP and AlAs presented in this thesis falls in the field of optical and luminescent properties, and is based on the following four publications:

- I Low Temperature Luminescence of GaN,
J Appl Phys 41, 4054 (1970)
(together with H G Grimmeiss).
- II Determination of Bandgap and Refractive Index of AlP from
Optical Absorption,
Solid State Commun 8, 1295 (1970).
- III Some Optical Properties of AlAs,
Solid State Commun 8, 15 dec 1970.
- IV Temperature Dependence of the Refractive Index for
AlAs and AlP,
to appear in Phys Status Solidi (a) 5, april 1971.
(together with H G Grimmeiss).

In the following a review of this work is presented, and an attempt is made to relate the results obtained by the author with those from other investigations relevant to this topic and published during the last few months.

III LOW TEMPERATURE LUMINESCENCE OF GaN AND ITS RELATION TO KNOWN OPTICAL PROPERTIES

In paper I results are presented from measurements on photoluminescence at low temperature (1,5K -150K) for GaN. At the time this investigation was performed, the only information in literature of any physical properties of GaN at temperatures lower than 90K was some questionable measurements of super-conductivity on GaN powder. Thus the situation called for an original work on the low temperature behaviour of this compound with regard to optical phenomena. The main conclusions drawn from the experiments reported in paper I concern the physical origin of the different emissions observed, and at the same time estimations for the bandgap and an LO-phonon energy, which were previously not known for this compound. After paper I was submitted for publication results from other measurements on some optical properties and even luminescence in GaN have appeared during the fall of 1970. Thus Pankove et al⁸⁾ measured luminescence from epitaxially grown layers of GaN between 2K and 300K under both UV-laser and 20 keV electron excitation. They have found a peak at 3,477 eV at low temperature, which ought to be identical with the "edge emission" in paper I, although Pankove et al report a considerably larger half width for this emission. This large half-width of 150 meV, compared with 7 meV in paper I, seems hard to explain unless considerable internal strain or other crystal defects are present in their samples to disturb the ideal shape of the absorption edge. The temperature dependence of this emission reported in ref 8 is also in agreement with that from paper I, and is believed to reflect the corresponding shift of the bandgap. No detailed study of the behaviour of the edge emission under different excitation conditions are presented in ref 8, probably due to the large half-width, and also no interpretation of any spectra are presented by

these authors, except for a structure at 3.37 eV at 2K which is believed to be connected with a bound exciton recombination. This 3.37 eV-emission is not present in our measurements.

According to the interpretation of the "edge emission" as a free exciton recombination in paper I, an estimation of the "exciton gap" $E_{ex} = E_g - 13.6 \frac{m_r^2}{\epsilon^2}$ (eV) as $E_{ex} \approx 3.48$ eV at low temperature was made. At the time paper I was submitted, no information about dielectric constant or refractive index of GaN was available. This situation is now somewhat changed, since several suggestions for the refractive index evaluated from experiments were presented during the last months^{5,8,9)}. The most convincing value for $\epsilon_\infty = n^2$ might be 5.8, as proposed by both Manchon et al⁹⁾ and Pankove et al⁸⁾. For an estimation of m_c^x we can make use of Kane's theory¹⁰⁾

$$\frac{1}{m_n^x} = 1 + \frac{2}{3} \frac{|P|^2}{\hbar^2} \cdot \left(\frac{2}{E_g} + \frac{1}{E_g + \Delta_0} \right).$$

Extrapolating the momentum matrix element $|P|$ from other III-V-compounds, we get the value $m_n^x \approx 0,13$ for the effective mass of electrons and estimate $m_p^x \approx 0,6$ ⁵⁾ for the holes at the lowest band edges. (The valence band spin-orbit splitting is assumed to be negligible for GaN¹¹⁾). Using the high frequency value $\epsilon_\infty = n^2$ for ϵ , a binding energy of about 0,04 eV is found from these values, and a value for the lowest bandgap E_g of about 3,52 eV at low temperature is anticipated.

This estimated value is partly in contrast with conclusions drawn from measurements on optical absorption at 300K for GaN-films published during 1970.^{4,5,12)} Collecting data from these three papers, a somewhat confused picture of possible bandgap estimations ranging from 3,39 eV⁴⁾ to about 3,96 eV⁵⁾ comes to light. From fig 1 of this thesis it is obvious, that in the present state no conclusions

at all should be drawn from these absorption measurements concerning the situation of the bandgap (or exciton edge). The authors apparently have not related the shape of the absorption edge at 300K to the free electron concentration n in their samples. According to Casella¹³⁾ the existence of excitons is possible only if the Debye screening length $L = \sqrt{kT\epsilon_0\epsilon/n e^2}$ is much larger than the exciton Bohr radius $a_{ex} = \frac{4\pi}{e^2} \cdot \frac{\epsilon_0\epsilon\hbar^2}{m_r}$. For GaN this means, that exciton effects are negligible if the free carrier concentration exceeds a value of about $5 \cdot 10^{17} \text{ cm}^{-3}$. The value of n for the samples used by Pankove et al¹²⁾ is stated to be about 10^{19} cm^{-3} , and therefore their "absorption knee" at 3,5 eV at 300K should correspond to transitions for energies above the bandgap E_g . One might also guess that the films prepared by various techniques contain a large number of lattice defects and internal stresses, which are known to broaden the absorption edge and even cause a shift to higher energy of the "absorption knee" generally referred to the direct gap exciton edge.^{14,15)} It is a wellknown fact, that the absorption edge of an amorphous semiconductor generally occurs at an appreciably higher energy than for a crystalline material¹⁶⁾ and there seems to be a graded shift for this "absorption edge" between these two limit states.¹⁴⁾

All the above authors^{4,5,12)} seem to agree that the bandgap should be direct, which had also been inferred from bandstructure calculations based on dielectric theory¹⁷⁾. A direct bandgap for GaN would support the interpretation of the "edge emission" in paper I, as a free exciton recombination. A definite test of this interpretation can only be made when reliable absorption and reflexion measurements made at low temperature on good single crystals are made, so that the expected exciton structure can be resolved. No such investigations seem to have been published to date. It is further worth men-

tioning, that a determination of the bandgap from diffuse reflexion measurements on powder samples of the same type as used in paper I, give a value for the bandgap E_g , at low temperature, as 3,45 eV¹⁸⁾. The uncertainty in E_g -values from this method is generally found to be better than 0,1 eV, as judged from measurements on other materials. The sharp rise observed in excitation spectra (fig 7 of paper I) seems also to allow a rather limited variation of the bandgap from the value predicted here. The value $E_g = 3,25$ eV¹⁾ previously used in textbooks is thus significantly lower than the correct one.

Turning to the so called satellite emissions, the first two of these were probably also seen by Pankove et al,⁸⁾ but they mentioned nothing about their possible origin. In paper I these emissions were tentatively attributed to a pair emission accompanied by LO-phonon replica. The LO-phonon energy of 90 ± 3 meV anticipated from this interpretation is in agreement with the values for LO-phonon energies reported by Manchon et al⁹⁾, considering the quoted uncertainties in their values. This is thus a support of the phonon replica model for these emissions instead of the old model¹⁾ involving several impurity levels.

The satellite emissions of GaN are believed in paper I to be of a similar origin as the so called "edge-emission" in II-VI-semiconductors such as e g CdS and ZnS. In this field a paper has recently appeared¹⁹⁾, which seems to shed new light over the possible origin of these emissions. From measurements on CdS, including investigations on band shape and polarisation of these emissions, the authors concluded, that even the high energy series (HES) of the green emissions generally observed is caused by a donor-acceptor-pair mechanism; in this case the pairs should be of a nearest neighbour type. The HES shows no shift with excitation density, but this

is not expected for a nearest neighbour transition. The low energy series (LES) in CdS is explained as due to pairs with larger separations and there the observed intensity shift is expected. If we compare these observations with those for the satellite emissions in GaN which did not show a significant shift with excitation density, we find a further support for the likeliness that a pair mechanism is the explanation for these broad emissions. The peaks observed might thus be dominated by nearest neighbour pairs. The odd asymmetry of these emissions (the emission profile seems to be decreasing slower at the low energy side) could also be explained as an unresolved tail due to more distantly spaced pairs. The width of the emissions could be explained by interaction with acoustic phonons, as indicated for CdS¹⁹⁾. The knowledge of the defects responsible for the emissions is of course very incomplete. One could guess that the origin is native defects in the crystals as for the II-VI-compounds, and that these might be related to the wellknown nitrogen deficiency in GaN³⁾.

IV OPTICAL ABSORPTION IN AlP

Measurements on optical absorption in AlP crystals are reported in paper II for temperatures 4K - 300K. These investigations were motivated by the considerable scatter in values for the forbidden band gap E_g reported earlier. Reliable optical measurements on this compound can only be made, if the crystals are protected against moisture, since otherwise an oxide layer forms quite rapidly, which tends to disturb the measurements. It was found, that an oxide layer of about 1-2 μ thickness is formed in a few minutes, when the crystals were exposed to normal air and after a day or two they were reduced to white powder. In spite of great care taken during crystal growth to transfer the crystals safely into evacuated ampullas, a thin oxide film estimated to a few tens of a micron was evidently present on all crystals, which made reliable UV-reflexion measurements impossible. Thus the optical investigations had to be restricted to transmission measurements, and due to a special preparation technique, the samples could be mounted in the cryostat without any exposure to air. A specially constructed glovebox was evacuated to high vacuum, and was then filled with dry helium gas when the ampullas were opened and the samples mounted. During the transfer to the cryostat the sample was also protected in dry helium atmosphere with a special valve. Thus a thick oxide layer, which might introduce errors in transmission e g via interference effects in the oxide layer, could be avoided.

At the time paper II was prepared, no other detailed measurements on optical properties of AlP had been published. After this paper was submitted for publication, Lorenz et al²⁰⁾ published similar measurements on AlAs and AlP. These authors had larger crystals available and could therefore measure the low level absorption near the edge with better accuracy. They were not either able to resolve

any reliable phonon structure at the edge for AlP and therefore the uncertainty of the values quoted for E_g is given to some hundreds of an eV. Their values for the bandgap E_g are 2,45 eV at 300K and 2,52 eV at 4K, which both lie within the limits proposed in paper II. These measurements thus agree regarding bandgap and type of transition. The lower value quoted in paper II might be due to a higher impurity concentration in our crystals. This possibility of band-shrinking was also pointed out in paper II.

An estimation for the direct gap in AlP from luminescence measurements on $Al_xIn_{1-x}P$ -alloys was recently presented by Onton et al²¹⁾. Their value, quoted from a linear extrapolation, is 3,6 eV, which is in agreement with the statement in paper II that the direct bandgap $\Gamma_{1C}-\Gamma_{15V}$ is larger than 3,1 eV.

For the refractive index of AlP no measurements other than those in paper II are known to the author to this date. It seems not possible to make an "a priori" estimation of the refractive index n of a semiconductor, without an experimental knowledge of related properties, such as e.g static dielectric constant. However, it is often argued that the value of n in non-elemental semiconductors is correlated to the ionicity of these compounds; the problem is, of course how to define ionicity. Following the simple one-gap model used by Van Vechten²²⁾, we can define an ionicity factor f_i as $f_i = \frac{C}{C+E_h}$; where E_h is the homopolar energy gap in this model²²⁾ and C is the heteropolar gap ($C \approx 0$ for elements). If we take Van Vechtens values for E_h ²²⁾, we get for GaP a value $f_i = 0,41$ ($n_o \approx 3,0$) and for GaAs $f_i = 0,40$ ($n_o \approx 3,3$). Using the low frequency value $n_o = 2,75$ from paper II, we get for AlP $f_i = 0,44$. Thus the lower value of n_o in AlP compared to GaP and GaAs might be expected.

V OPTICAL PROPERTIES AND LUMINESCENCE IN AlAs

Similar measurements are reported in paper III for AlAs, as those for AlP in paper II, but here low temperature luminescence is also included. Reliable absorption measurements for AlAs down to 2K were already published during the preparation of paper III²⁰⁾ and therefore the main results from transmission measurements published in paper III are those concerning higher absorption levels and refractive index. Although the values of α above 3,0 eV are uncertain, the results clearly show, that the direct bandgap $\Gamma_{1C} - \Gamma_{15V}$ is significantly higher than 2,9 eV at 300K previously reported by several authors^{23,24)}. Thus the theories for the composition dependence of the direct gap in $Al_xGa_{1-x}As$ also have to be reconsidered.²⁴⁾

Very recently the author was informed about (as yet unpublished) measurements on optical absorption in AlAs performed by W M Yim at RCA Lab in Princeton.²⁵⁾ He reports a value of 3,14 eV at 300K for the direct bandgap $\Gamma_{1C} - \Gamma_{15V}$, however, his measurements were performed in air and influence of the oxide layer was not considered.

The value for the refractive index $n_0 = 2,9$ for AlAs found in paper III is in reasonable agreement with estimations in other work done at the same time^{20,26,27)}, however a letter appeared recently²⁸⁾, where a value of 3,9 was reported from IR-reflexion measurements on AlAs-layers deposited on GaAs. This is in sharp disagreement with the value from paper III. If this value $n_0 = 3,9$ is tentatively applied to the low energy cut off of the interference pattern in transmission for the crystals measured in paper III, to adjust the absolute interference order p in the formula $2nd = p\lambda$, then the resulting dispersion curve for n is decreasing with rising photon energy, and n amounts to about 3,6 at 2,5 eV, which indeed would be an anomalous behaviour. This curve is shown in fig 2 of this

thesis for a typical crystal. Mindens value is also in disagreement with the accepted theory for the superior performance of the "close confinement" laser structures in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ ²⁹⁾. Since only one crystal seems to have been measured by Minden, and considerable stratification is observed in the deposited layers²⁸⁾, it might be possible that the thickness of the reflecting part of the lamellæ somehow was incor^rrectly determined.

Apparently other measurements, which are yet unpublished, exist on the refractive index n of AlAs²⁵⁾. Thus G D Pettit has used the prism method to determine n at 6328Å, and a value of 3,09 is reported, in fair agreement with the corresponding value of $3,05 \pm 0,13$ from fig 1 in paper II. Further a value of $n = 2,85$ in the infrared has been communicated by R E Fern²⁵⁾, to be compared with $n = 2,90 \pm 0,13$ in paper III. Thus it seems likely, that the correct value for the refractive index in AlAs will be covered by the results reported by the author in paper III. Raman spectroscopy work recently communicated by A Onton³⁰⁾ have accurately established the zone center optical phonon frequencies of AlAs as $\hbar\omega_{\text{LO}}(\Gamma) = 50,1 \text{ meV}$ and $\hbar\omega_{\text{TO}}(\Gamma) = 44,8 \text{ meV}$. From these values, and the Lyddane-Sachs-Teller relation³¹⁾ $\epsilon_0 = n^2 \cdot \left(\frac{\omega_{\text{LO}}}{\omega_{\text{TO}}} \right)^2$, we can estimate a value of the relative static dielectric constant ϵ_0 as $\epsilon_0 = 10,5$, subject to experimental verification.

The luminescence measurements at low temperatures were also original measurements, since no reports on luminescence of AlAs in the temperature region 1,6K-77K were found in literature when this work was completed. The results obtained seem to support the assumption that AlAs, even with respect to luminescence, behaves very much like GaP, which has been extensively studied during the last 10 years. After the submission of paper III, two papers have appeared dealing

with luminescence in AlAs. The first one, by Kressel et al ³²⁾, reports on photo- and cathodoluminescence in AlAs down to 7K and several emission peaks were found. However, very little qualitative comparison with paper III can be made, since the spectra shown in ref 32 are entirely different from those in paper III, presumably due to different impurities incorporated in the crystals. The most dominant peak at 7K reported in ref 32 is believed to be due to donor-acceptor pair recombinations, in agreement with the author's observations at the lowest temperatures. Luminescence in AlAs was also measured by Bindemann et al ³³⁾, but their experiments were not carried further down than to 27K. Therefore these spectra are not either quite comparable with those in paper III, although a pair mechanism is suggested also in ref 33 for the most high-energetic emissions observed. The purity of the crystals used by these authors was probably rather bad, since the impurity-induced band shrinkage was extremely large ³⁴⁾ compared with our samples and those of Lorentz et al ²⁰⁾. It is obvious that the interpretation of the chemical origin of the emissions observed will have to wait, until impurity content can be better controlled in the process of crystal growth. One conclusion that might be drawn from paper III and ref 32 is that the shallow optical ionization energies for donors and acceptors are at least of the order 0,05-0,06 eV. From earlier Hall-measurements on AlAs a value of 18 meV was suggested for the thermal ionisation energy for a Si donor ³⁵⁾. This value seems far too low, if we consider the above discussion of the dielectric constant in AlAs.

VI TEMPERATURE DEPENDENCE OF REFRACTIVE INDEX IN SEMICONDUCTORS

Paper IV deals with a new topic in semiconductor physics which was not even qualitatively covered before, namely the temperature dependence of the refractive index. The measurements presented are the first published for any semiconductor covering the whole temperature range 0K - 300K, and therefore they are of very general interest, although they were carried out only for the two "exotic" III-V-compounds AlP and AlAs. These investigations are therefore believed to stimulate the interest in similar work for other semiconductors, especially those where the physical properties are already well known, and thus comparison with theory can readily be made.

Older measurements for the temperature coefficient $\frac{1}{n} \frac{dn}{dT}$ were often related to a simple theory proposed by Moss³⁶⁾, which relates the refractive index to the smallest bandgap E_g of the material by the semiempirical formula $n^4 \cdot E_g = \text{const.}$ This formula applies out quite well for the temperature coefficients $\frac{1}{n} \frac{dn}{dT} = \frac{1}{4} \frac{dE_g}{dT}$ at room temperature for a number of semiconductors, but there are exceptions^{36,37)}. Further it might be argued, that the smallest bandgap in these indirect semiconductors should not be the most important parameter in connection with fundamental optical properties. This might be the reason why Yu and Cardona³⁸⁾ (YC) recently presented a new approach to the theory for the temperature dependence of the refractive index in semiconductors. This theory is based on a simple isotropic one-gap model for the bandstructure of a semiconductor devised by Penn³⁹⁾ in 1962. Due to its simplicity, the Penn model has been extensively used in calculations of various properties of semiconductors³⁸⁾. The theory for the temperature dependence of the long wavelength refractive index referred to here³⁸⁾, was

developed from a calculation of Haine and Jones⁴⁰⁾ for the pressure coefficient of the dielectric constant in a diamond type semiconductor. In the extension of this theory to the III-V-compounds, several simplifications were made by YC. Thus the antisymmetric pseudopotential coefficient $V_g(200)$ is neglected, which generally seems to be justified according to the bandstructure calculations reported by Cohen and Bergstresser⁴¹⁾, however these coefficients are not known for AlAs and AlP. Further in the calculation of the temperature dependence of the Debye-Waller factor, the average square displacements $\langle u_{\text{III}}^2 \rangle$ and $\langle u_{\text{V}}^2 \rangle$ for the atoms in the third and fifth column respectively are put equal. This might introduce some error for AlAs, due to the large mass ratio between Al- and As-atoms. The approximations made by YC in calculating the volume coefficient of the "Penn gap" $\hbar \omega_g$ are not serious, since the corresponding term contributes less than 10% to the temperature coefficient of the refractive index of AlAs and AlP. Thus these simplifications made in the theory should not introduce a larger error than 10% in $\frac{1}{n} \frac{dn}{dT}$ for these two compounds; bearing in mind the crudeness of the "Penn model" underlying these calculations. The most important source of error in the theoretical calculation should thus be the uncertainty in the Debye temperatures Θ_D for AlAs and AlP, since only extrapolated values at 0K are available⁴²⁾ and no experimental data. The good agreement between theoretical values for $\frac{1}{n} \frac{dn}{dT}$ at 300K and those found from experiment in paper IV might however indicate, that our estimated values for Θ_D are close to the correct ones.

Whether the theory used is applicable for all temperatures cannot be settled until we know more about thermal and optical properties of AlAs and AlP. Similar measurements as those performed by the author should also be done on more "conventional" semiconductors, where all parameters are experimentally known. The results obtained

in paper IV however, certainly do not constitute an objection to the theoretical approach, although a more refined model might be needed to get an exact fit to experimental values.

VII SUMMARY

It is clear from the exposure made in this thesis, that the interest in the three III-V-compounds GaN, AlAs and AlP has risen remarkably during the last two years, compared with the very few results published during two decades before. It might thus be expected that the fundamental properties of these semiconductors will be explored as well as our "conventional" semiconductors mentioned in the introduction. It is certain that many important applications for optical and electronic devices can be expected for these large band-gap compounds.

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FIGURE CAPTIONS

- Fig 1 Absorption spectra at 300K for GaN films published in ref 4,5 and 12.
- Fig 2 Dispersion of refractive index for AlAs according to paper III (lower curve) and ref 28 (upper curve). The upper curve is calculated using the value $n = 3.90$ to get the interference order at 0,6 eV, and then the same interference spectrum in transmission which was used for the lower curve.

